



Office de la propriété
intellectuelle
du Canada

Un organisme
d'Industrie Canada

Canadian
Intellectual Property
Office

An Agency of
Industry Canada

*Bureau canadien
des brevets
Certification*

*Canadian Patent
Office
Certification*

La présente atteste que les documents
ci-joints, dont la liste figure ci-dessous,
sont des copies authentiques des docu-
ments déposés au Bureau des brevets.

This is to certify that the documents
attached hereto and identified below are
true copies of the documents on file in
the Patent Office.

Specification and Drawing, as originally filed, with Application for Patent Serial No:
2,335,745, on February 13, 2001, by **HER MAJESTY THE QUEEN IN RIGHT OF
CANADA AS REPRESENTED BY THE MINISTER OF THE DEPARTMENT OF
FISHERIES AND OCEANS**, assignee of Dave Higgs, Bob Cairns and Ian Shand, for
"Process for Preparing Nutritionally Upgraded Canola Products". The said invention was
made while Dave Higgs was employed as a public servant, as defined in the Public
Servants Inventions Act in the Department of Fisheries and Oceans, pursuant to Section 5
of that Act, the said invention has been determined to be vested in **Her Majesty The
Queen In Right Of Canada As Represented By The Minister Of Fisheries and
Oceans**.

**CERTIFIED COPY OF
PRIORITY DOCUMENT**

Gray Laithwaite
Agent certificateur/Certifying Officer

May 25, 2005

(Date)

Canada

(CIPO 68)
31-03-04

OPIC  CIPO

ABSTRACT

A process for producing nutritionally upgraded canola protein products for use in fish or other animal diets. The process also allows for the production of novel canola oils as well as products suitable as components in organic fertilizers. In the process, raw and dehulled canola seed is subjected to rapid heat treatment in order to deactivate, destroy or reduce the concentration of at least some of the antinutritional components present in the oilseed; dehulled; cold pressed; and then subjected to solvent extraction in order to reduce the lipid content of the resulting meal product which may be used as is or further subjected to additional steps including blending, cooking, pressing, drying and condensing steps. Alternatively and depending upon the regional market requirements, the raw and dehulled canola seed is cold pressed and directly subjected to the above mentioned additional steps. Products produced by the process are also included.

PROCESS FOR PREPARING NUTRITIONALLY UPGRADED CANOLA PRODUCTS

5 FIELD OF THE INVENTION

The present invention relates to animal feeds, including fish feed. More specifically, it relates according to one embodiment to a process for producing nutritionally upgraded canola protein products for use in fish or other animal diets; other embodiments of the present invention relate to novel canola protein
10 concentrates and novel protein and lipid-rich canola meals, as well as novel canola oils produced using the process of the present invention.

BACKGROUND OF THE INVENTION

15 Feed accounts for about 35-60% of the operating costs of salmon farms and represents the largest cost in the culture of other carnivorous aquatic species. Moreover, the protein and lipid sources presently account for the majority of the feed cost. Accordingly, salmon farming is marginal in many regions. Hence, there is a need to reduce production costs and improve the market value of the farmed
20 product.

The use of less expensive alternative protein and lipid sources has been considered to reduce the cost of the protein and lipid sources in salmon feeds. One approach is to use protein and lipid sources that are based on processed-oilseeds rather than fish meal and oil. The plant oils need to be highly digestible with
25 appropriate fatty acid compositions and the plant protein products need to be in the form of nutritionally upgraded meal, protein concentrates, or possibly isolates. To date, most research on oilseeds has focused on the use of products derived from processing soybeans, rapeseed/canola, sunflower seed, cottonseed, and the like.
30 Comparatively few of these studies, however, have been directed to the feasibility of using canola. Indeed, although proteins contained in canola are rich in lysine

and methionine, both of which are limiting amino acids in most cereal and oilseed proteins, the use of canola as a protein source in food products has been severely limited, due to the fact that the proteinaceous material which is left over after oil is extracted by known processes contains antinutritional constituents. The latter include insoluble and soluble fibres, glucosinolates (antithyroid compounds), phenolic compounds and phytic acid.

It has been shown that the concentrations of the above mentioned unwanted constituents should be minimized in order to allow full expression of the high quality of canola protein and to improve the overall digestibility, palatability, as well as bioavailability of minerals in the canola protein product not only in terrestrial species but also in aquatic species as well.

U.S. Patents No. 4,233,210 to Koch and No. 4,889,921 to Diosady et al. disclose preparations of protein concentrates for use in animal or human nourishment, from oilseeds including rapeseed (canola). The various processes of these inventions generally comprise heating, drying and distillation steps, as well as treatments with alkaline solutions and extractions with organic solvents.

The protein extract claimed by Cameron et al. in U.S. Patents Nos. 4,418,013 and 4,366,097; and by Murray et al. in U.S. Patents Nos. 5,844,086 and 6,005,076 is said to be "protein isolate", which is regarded as being different from a protein concentrate. Indeed, it is established that a protein extract is an isolate when the protein content exceeds 90% and the protein is undenatured. Accordingly, the process of the preparation of an isolate does not allow for a heating step at elevated temperature.

Lawhon et al. in U.S. Patent No. 5,086,166 disclose a process allowing for the simultaneous preparation of protein as precipitate or curd, and oil for use as food products or food ingredients, from numerous oilseeds including rapeseed (canola). At an early step of the process, a heating treatment (at about 60°C to

90°C) of the material in water is performed, in order to inactivate enzymes inherent in the seed.

5 A process for the preparation of rapeseed and canola protein concentrates known as the "FRI-71 process" has been described by Jones (J. Amer. Oil Chem. Soc. 56, 1979, 716-721). This process allows for the production of highly digestible protein concentrates with reduced levels of antinutritional factors (except for phytic acid) that can be used to entirely replace the fish meal portion of diets for trout. However, subsequent work conducted in collaboration with the POS Pilot Plant Corporation in Saskatoon revealed that the FRI-71 process was not cost effective, due to low yields of the concentrates, and insufficient numbers of other value-added products apart from canola oil stemming from the process. Also, the process as described could not easily be applied in the private sector using existing oilseed and fish meal processing technology.

15 In the present invention, a modified FRI-71 process is described that results, besides the high value canola protein concentrate and animal feed grade canola oil, in other value-added products such as canola oil suitable for the organic food market, nutritionally upgraded canola meal, and products suitable as components in organic or predominately organic fertilizers. The process of the invention is simple and economical. Moreover, the process is readily integrated into existing oilseed crushing plants or fish meal production plants.

25 An object of the invention for certain embodiments is to provide an improved process for extracting protein and oil (human and animal feed grade) from canola. A further object of other embodiments is to provide protein products that are particularly well suited for use in high energy (lipid) diets for fish farming and in some animal feeds.

SUMMARY OF THE INVENTION

One embodiment of the invention is directed to a process for producing nutritionally upgraded canola meal and/or concentrates with low levels of antinutritional constituents (except phytic acid) and reduced fibre content (in some cases), for use in fish or other non-human animal diets. Also in another embodiment of the invention, there are produced canola oils (for organic and general human food, as well as animal feed markets) and products suitable as components in organic fertilizers.

The process consists of either cold pressing (temperature $<85^{\circ}\text{C}$) raw and undehulled canola seed and then following steps i-vi as described below or alternatively depending upon regional market requirements following steps 1-4 and then i-vi. Steps 1-4 include:

- (1) subjecting canola to rapid heat treatment in order to deactivate, destroy or reduce the concentration of at least some of the antinutritional components present in the oilseed and facilitate dehulling;
- (2) dehulling (preferably partially) the heat-treated seed through an impact disc or other appropriate mechanical process;
- (3) cold pressing the resulting meats to yield an oil suitable for the organic human food market and a meal containing about 30-33% protein and about 30-38% lipid, with reduced fibre content; and optionally
- (4) reducing the lipid content in the meal when required through hexane extraction and recovering the solvent.

The resulting meal product may be used as is as a component of a high energy diet for fish or other farmed animals, or the resulting meal (without any removal of lipid) may be further processed as follows to yield a canola protein concentrate and other derivatives as described below. The additional steps (i-iv) include:

- (i) blending the meal with a suitable amount of water (~5-8:1 w/w; water to oil-free dry matter content of the meal) and an antioxidant;

(ii) cooking the mixture at about 90-93°C with water in order to improve protein digestibility and free the cellular lipid that remains in the meal;
(iii) pressing (screw press or expeller press) and/or centrifuging the mixture to yield solid and liquid fractions and then separating the liquid fraction by continuous centrifuge into stick water and oil (animal feed grade unless the oil is subjected to additional processing steps such as degumming, alkali refining, bleaching, etc. to yield a human grade oil);
(iv) optionally submitting the protein fraction to a hexane extraction if its lipid content is too high, with subsequent recovery of the solvent and the oil;
(v) drying the protein fraction using a low temperature (about 75-83°C) process to yield a high value canola protein concentrate; and
(vi) condensing the stick water fraction to about one third of its original volume and then directing the condensed acid stabilized solubles along with the hulls for use as components in organic fertilizers.

In accordance with another embodiment of the invention, the process alternatively involves the steps of:

(i) subjecting canola seed to cold pressing under conditions selected to substantially deactivate, destroy or reduce the concentration of at least some of the antinutritional components normally present in canola seed to produce pressed raw seed;
(ii) blending said pressed raw seed with water and an antioxidant;
(iii) cooking the mixture of said pressed raw seed, water and antioxidant at about 90-93°C;
(iv) separating the mixture into stick water fraction, protein-rich fraction, and oil fraction; and
(v) drying said protein-rich fraction to yield high value canola protein concentrates suitable for use in animal diets.

In accordance with other embodiments of the invention, there are provided canola protein concentrates prepared by the process noted above, that contain

from about 50% to about 68% protein, that are highly digestible. These concentrates are also rich in digestible energy content especially if they originate from the processing of dehulled canola and they are significantly reduced in antinutritional constituents except for phytic acid. The canola protein concentrates of the present invention also have moderate contents (from about 7% to about 12%) of lipid that are extensively comprised of highly digestible monounsaturated and polyunsaturated fatty acids.

Another embodiment of the invention is to provide canola oils as by products of the process described above, that are suitable for the organic and regular human food market and animal feed market.

In another embodiment of the present invention, there is provided a method for recycling the condensed solubles stemming from the treatment of canola seed to produce the protein concentrates and the canola oils. Said method consists of directing the condensed solubles together with hulls for use as components in organic fertilizers.

In a further embodiment of the invention, there is provided a process allowing for the production (preferably simultaneously) of a nutritionally upgraded canola meal and protein concentrates, a human and animal feed grade canola oil and a mixture of products for use as components in organic fertilizers.

BRIEF DESCRIPTION OF THE DRAWING

Having generally described the invention, reference will be made to the following drawing illustrating preferred embodiments only. In the drawing:

Figure 1 is a schematic diagram of the process according to one embodiment of the invention.

DETAILED DESCRIPTION OF THE PREFERRED EMBODIMENTS

The steps involved in the process of the invention are illustrated in Figure 1. In this Figure, there is shown a schematic representation of the processing of canola seed to yield cold-pressed oil indicated as product 1; hulls from dehulled
5 meats indicated as product 2; nutritionally upgraded canola meal produced from heated, dehulled and cold pressed canola indicated as product 3; animal feed-grade oil indicated as product 4; condensed solubles indicated as product 5; and high nutritive value concentrated protein indicated as product 6. Undehulled oilseed (A) or dehulled seed B, preferably for monogastric diets) may be used. An
10 optional lipid extraction and solvent recovery step can be inserted just before or after press cake drying and to reduce the lipid content of canola meal.

The initial step involves cold pressing (temperatures $<85^{\circ}\text{C}$ using a suitable cold press) raw whole canola seed or alternatively subjecting the raw seed to rapid
15 heat treatment and then dehulling and cold pressing. If the latter option is selected, one procedure involves heating the seed at $110-115^{\circ}\text{C}$ for 90 seconds followed by 30 min at $100-110^{\circ}\text{C}$. Other options require less heat depending upon the form of heat and whether or not a vacuum is applied during the heat process. The
20 temperature and length of the treatment is selected to substantially: (i) deactivate or destroy the activity of myrosinase, which is the enzyme responsible for glucosinolate hydrolysis; (ii) improve the digestibility or bioavailability of the carbohydrates present in canola and (iii) reduce the moisture content in the seed, which results in a partial separation of the meat from the fibrous indigestible hull. The dehulling process is further completed by subjecting the heat-treated seed
25 through an impact, a disc, or other mechanical process coupled with a gravity screening or air-classification process.

The canola meats that result from the cold pressing of the raw, unheated seed are not marketed directly for use in high energy animal feeds, unlike those
30 originating from the cold pressing of heated, dehulled seed which have been nutritionally upgraded due to their reduced content of fibre and one or more

antinutritional factors. Accordingly, the latter meal which contains about 30-33% protein and 30-38% lipid, is used as is or it is hexane extracted to reduce its lipid content further and concurrently elevate its protein concentration with recovery of solvent before channelling it directly into diets of aquatic and terrestrial species, or similar to the meal from the unheated, pressed seed, it is submitted without lipid extraction to the next step of the process. The cold pressed oils from both sources, however, are channelled into the high value organic human food market.

The meals from undehulled or dehulled canola are blended with a suitable amount of water (~5-8:1 w/w water to oil-free dry matter of meal) and an antioxidant (e.g. 100 mg of santolquin/kg of meal). The added water serves to wash the canola meal as the blend moves through the cooker to either a continuous screw press that is surrounded with perforated screens or an expeller press. As the presscake moves through this stage of the apparatus, fluids are drained off that include water that contains soluble protein, some of the remaining water soluble antinutritional components such as glucosinolates, phenolic compounds and unwanted sugars like raffinose and stachyose; as well as a large portion of the lipid fraction. The mechanical separation of the aforementioned solids and liquid fractions may also involve the use of a decanter centrifuge depending upon the efficiency of liquid/solid separation after the presscake has passed through the screw press or expeller.

Thereafter, the fluid mixture is separated by continuous centrifuge into stick water and animal feed grade oil fractions (the latter may be subjected to additional processing steps as referred to previously to create a human grade oil). The presscake meal is dried using a low temperature process (temperature of about 75°C to about 83°C) to yield a dried protein-rich fraction (concentrate).

In cases where the lipid content of the dried protein fraction is too high for the desired animal feed use, a solvent extraction step involving hexane is performed, with subsequent recovery of the solvent and the animal feed grade oil.

In another embodiment of the invention, the solvent extraction step is performed prior to the low temperature drying step.

5 The stick water fraction mentioned above is condensed to about a third of its original volume and following acid stabilization, is then used together with the hulls as components in organic fertilizers for agriculture.

10 Given the above teachings, it will be seen that the invention also provides a protein concentrate produced by the above process, containing from about 50% to about 68% protein, that is highly digestible and significantly depleted in antinutritional constituents (except for phytic acid) that were present in the original canola oilseed. The canola protein concentrate of the present invention has a moderate content (from about 7% to about 12%) of lipids including highly digestible monounsaturated and polyunsaturated fatty acids.

15

20

25

30

WE CLAIM:

1. A process for preparation of nutritionally upgraded canola meal and high value canola oil from canola seed for use in fish or other non-human animal diets or in human foods comprising the steps of:
 - subjecting said canola seed to heat treatment under conditions selected to substantially deactivate, destroy or reduce the concentration of at least some of the antinutritional components normally present in canola seed to produce heat-treated seed;
 - dehulling said heat-treated seed to produce a meat fraction and a hull fraction; and
 - cold pressing said meat fraction to yield an oil suitable for use in the organic human food market and a protein and lipid-rich meal, with reduced fibre content.
2. A process for preparation of canola protein concentrates from canola seed for use in fish or other non-human animal diets comprising the steps of:
 - subjecting said canola seed to heat treatment under conditions selected to substantially deactivate, destroy or reduce the concentration of at least some of the antinutritional components normally present in canola seed to produce heat-treated seed;
 - dehulling said heat-treated seed to produce a meat fraction and a hull fraction;
 - cold pressing said meat fraction to yield a high value human grade oil and a protein and lipid-rich meal with reduced fibre content;
 - blending said protein and lipid-rich meal with water and an antioxidant;
 - cooking the mixture of said protein and lipid-rich meal, water and antioxidant at about 90-93°C;
 - separating the mixture into stick water fraction, protein-rich fraction, and oil fraction; and

- drying said protein-rich fraction to yield high value canola protein concentrates suitable for use in animal diets.

3. The process according to claim 1 or 2, wherein said protein and lipid-rich meal is further submitted to a solvent extraction, with subsequent recovery of the solvent and the oil.
4. A process for a preparation of canola protein concentrates from canola seed for use in fish or other non-human animal diets comprising the steps of:
 - subjecting said canola seed to cold pressing under conditions selected to substantially deactivate, destroy or reduce the concentration of at least some of the antinutritional components normally present in canola seed to produce pressed raw seed;
 - blending said pressed raw seed with water and an antioxidant;
 - cooking the mixture of said pressed raw seed, water and antioxidant at about 90-93°C;
 - separating the mixture into stick water fraction, protein-rich fraction, and oil fraction; and
 - drying said protein-rich fraction to yield high value canola protein concentrates suitable for use in animal diets.
5. The process according to any one of claims 1 to 3, wherein said heat treatment is performed at a temperature of about 100-115°C.
6. The process according to any one of claims 1 to 3, wherein said heat treatment is performed at a temperature lower than 100°C in the presence of a vacuum.
7. The process according to any one of claims 1 to 3, wherein said dehulling is a partial dehulling step.

8. The process according to any one of claims 1 to 7, wherein said cold pressing is performed at a temperature not exceeding 85°C.
9. The process according to any one of claims 2 to 8, wherein said blending is performed in a horizontal mixer.
10. The process according to any one of claims 2 to 9, further comprising the step of observing a little delay prior to subjecting said mixture to said cooking step.
11. The process according to any one of claims 2 to 10, wherein said antioxidant is selected from the group consisting of ethoxyquin (santoquin), butylated hydroxyanisole, butylated hydroxytoluene, natural antioxidants and mixtures thereof.
12. The process according to any one of claims 2 to 11, wherein said separation of said mixture is performed by screw press, expeller press or decanter centrifuge.
13. The process according to any one of claims 2 to 12, wherein said drying of said protein-rich fraction is performed using a low temperature process, at a temperature of about 75°C to about 83°C.
14. The process according to any one of claims 2 to 13, wherein said protein-rich fraction is further submitted to a solvent extraction, with subsequent recovery of the solvent and the oil.
15. The process according to any one of claims 1 to 14, wherein said solvent comprises hexane.
16. The process according to any one of claims 2 to 15, further comprising the

step of condensing said stick water fraction to yield condensed solubles.

17. A canola protein concentrate for use in fish or other animal diets, produced in accordance with any one of claims 2 to 16, containing from about 50% to about 68% protein and from about 7% to about 12% lipid.
18. A protein and lipid-rich canola meal for use in fish or other non-human animal diets, produced in accordance with any one of claims 1, 3 or 4 to 17, containing from about 30% to about 33% protein and about 30% to about 38% lipid.
19. A canola oil produced in accordance with any one of claims 1 to 18, suitable for human or animal consumption.
20. The process according to any one of claims 2 to 16, further comprising the step of using said condensed solubles together with hulls as components in organic fertilizers.

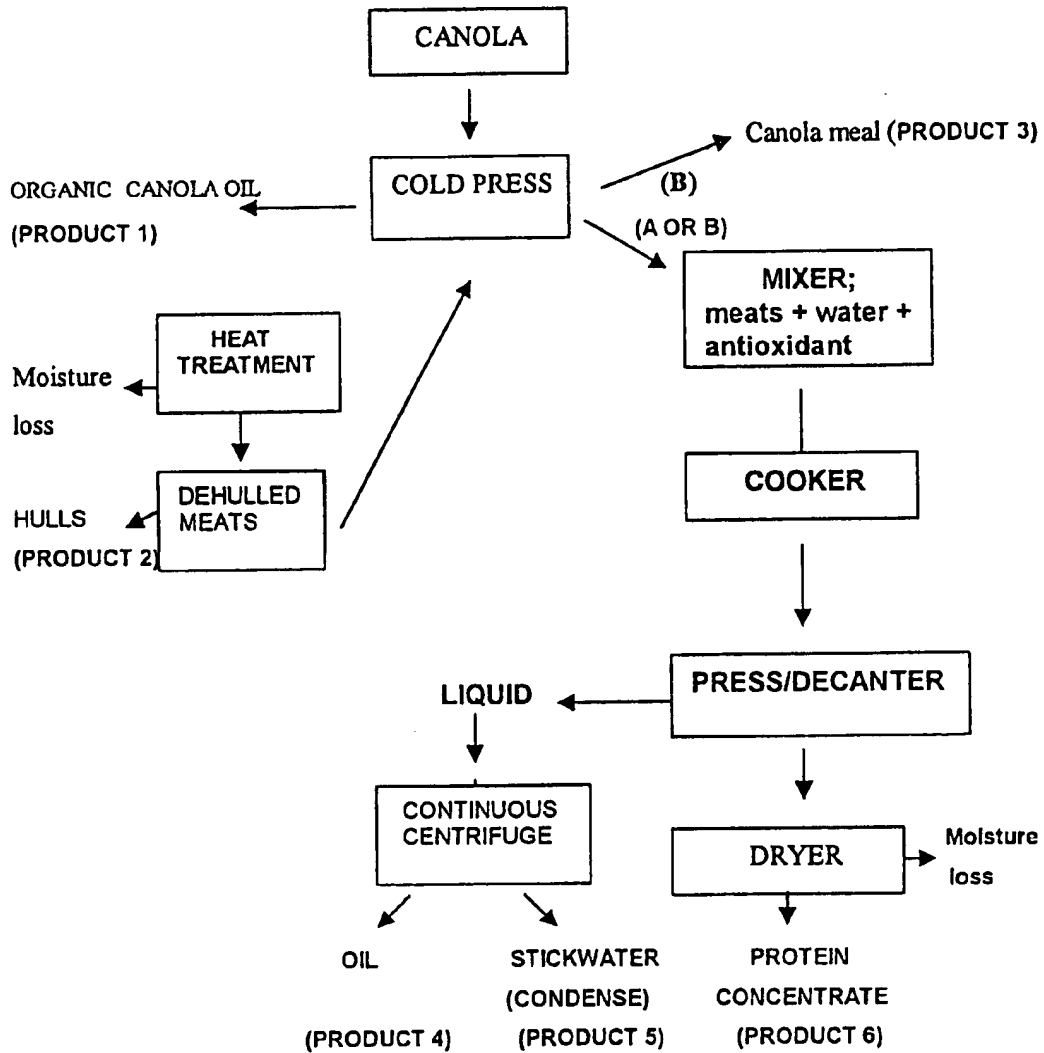


Figure 1